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APPLICABILITY OF THE KRYPTONATE® TECHNIQUE TO THE PREDICTION OF USEFUL FATIGUE LIFE

PHILIP GOODMAN

Panametrics, Inc.

TECHNICAL REPORT AFFDL-TR-69-55

JANUARY 1970

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APPLICABILITY OF THE KRYPTONATE® TECHNIQUE TO THE PREDICTION OF USEFUL FATIGUE LIFE

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FOREWORD

This research and development project was performed by Panametrics, Inc., Waltham, Massachusetts, under USAF Contract No. F33615-68-C-1124. The work was performed under the supervision of Dr. Philip Goodman, the author of this report. The contract was initiated under Project No. 1347, "Structural Testing of Flight Vehicles," Task No. 134702, "Measurement of Structural Responses." The work described in this document was authorized by and completed under the auspices of the Structures Division, Air Force Flight Dynamics Laboratory, Air Force Systems Command, Wright-Patterson Air Force Base, Ohio. It was funded by In-House Laboratory Independent Research Funds (Laboratory Director's Funds). Mr. Dwight Fearnow was the initial project engineer and he was succeeded by Lt. W. C. Eddy and Mr. Robert A. Noble, (FDTT).

This report covers work conducted from November 1967 to April 1969. It was released by the author in April 1969.

This technical report has been reviewed and is approved.

Robert L. Cavanagh

ROBERT L. CAVANAGH
Chief, Experimental Branch
Structures Division
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ABSTRACT

The behavior during stress cycling of a Kryptonate®, prepared from aluminum alloy 7075-T6 fatigue specimens, was studied. Kryptonates are solids into the surface of which the radioisotope, Kr^{85}, has been stably incorporated. They release Kr^{85} at a rate dependent upon the rate at which the surface is disturbed either physically or chemically. Their preparation and properties are discussed.

Efforts to determine whether the release rate of Kr^{85} from a fatigue specimen would be a predictor of remaining fatigue life are reported. Other properties of krypton impregnated specimens, such as high sensitivity to chemical attack and Kr^{85} thermal release behavior were investigated in relation to the fatigue process.

A correlation was found between the onset temperature of Kr^{85} thermal release and the extent of previous stress cycling. The validity of a fatigue life test based upon Kr^{85} loss behavior from aluminum alloy 7075-T6 fatigue specimens was established negatively.

Distribution of this abstract is unlimited

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1. Introduction

Fatigue failure, particularly in aircraft structures but in other structures as well, has been a severe problem and has received considerable attention and study. Prediction of fatigue life from load history has not been possible and hence costly inspection and rework have been required well before failure is imminent. An inspection procedure which could assess the extent of fatigue damage and predict the remaining useful life would therefore reduce inspection and rework costs.

The objective of the present program was to assess the applicability of the Kryptonate® * technique to the prediction of useful fatigue life. The Kryptonate technique has enjoyed remarkable success in a number of divergent areas (1) including trace gas detection and measurement, refractory metal oxidation studies, and surface temperature measurement. Kryptonates are solid materials into which radioactive Kr^{85} has been introduced and trapped. Because Kr is an inert gas, the physical and chemical properties of the solid remain unaltered. The release of Kr^{85} is easily detected with high sensitivity by standard radiotracer techniques and this release rate has been found to be dependent upon the rate at which a surface is disturbed either chemically or physically.

This program was undertaken to determine whether the release rate of Kr^{85} from a fatigue specimen would be a predictor of remaining fatigue life. These studies, discussed in Section 3, indicated that the loss rate was too small to be useful for this objective. The small loss rate is attributed to the fact that deformations involved in fatigue testing remain in the elastic region and therefore do not involve a disruption of the crystalline order. Other properties of Kryptonates, such as high sensitivity to chemical attack and Kr^{85} thermal release behavior, were investigated in relation to the fatigue process. These studies are discussed in Section 4.

2. Background

2.1 Preparation and Properties of Kryptonates

Kryptonates are solid materials which have been treated in such a manner as to incorporate radioactive Kr^{85} into their structure. The release of Kr^{85} from such solids has been found to be an extremely useful "tracer" to probe both chemical and physical changes occurring

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in the solids. At least two general methods, described in detail in reference (2), are available for preparing Kryptonates from solid materials. One is diffusion of Kr into the solid under conditions of relatively high temperature and high Kr pressure; the other is ion bombardment in which the gaseous Kr is ionized and then accelerated into the surface of the material by a high electrical potential. Either method results in trapping of Kr in the surface layers of the solid at depths ranging from 200Å to a maximum of about 1μ.

The behavior of solid Kryptonates has been found to be rather unique. In general, physical or chemical changes occurring at the surface of the host material result in the release of entrapped Kr. Thus, for example, the oxidations of graphite and copper surfaces have been observed by means of the release of Kr (2). This phenomenon has led to the development of an extremely sensitive technique for oxygen detection. Surface chemical attacks by other gases such as H_2 and F_2 (3) and by liquids (4) have been effectively probed by this method. Even mechanical removal of surface layers which occurs by wear has been detected and measured by the removal of Kr^{85} from the surface layers (5).

Another unusual property which has been observed is the effect of temperature cycling upon the degassing behavior of a Kryptonate. In general, when a solid Kryptonate is heated to any given temperature, some fraction of the Kr activity is lost. If the sample is maintained at this temperature, the rate of loss decreases until it is negligible, and the sample retains this activity level almost indefinitely. No significant loss of Kr will then occur as a result of thermal cycling unless the sample temperature exceeds the original temperature. If this should occur, the sample loses another fraction of its activity and then "equilibrates" at the new "conditioning" temperature. This property has been utilized to measure the surface temperature of metals subjected to unknown temperature environments. In particular, temperatures have been measured on jet and helicopter engine parts (5, 6) and on surfaces undergoing mechanical friction (5).

Mechanisms of retention of rare gas atoms by solid structures are not completely understood. It has been shown (7), however, that such rare gases are trapped by sites which are associated with grain boundaries as well as by sites which are intragranular. The binding energy, and hence the release temperatures, are higher for the latter type of site.

3. Initial Program

As stated earlier, the initial objectives of this program were intended to ascertain the correlation that exists between the rate of Kr^{85} loss from a fatigue specimen and the fatigue life of that specimen. A statistically valid testing program at several different stress levels was envisaged. Accordingly, a specimen, shown in Fig. 1, was designed suitable for use in a Schenk Model PVQ Vertical Fatigue Machine.

3.1 Specimen Preparation

Most specimens were machined from aluminum alloy 7075-T6 sheet stock but a few specimens of 7075-O alloy were obtained. All specimens were electropolished prior to Kryptonate preparation in order to minimize interference from surface effects.

Three different types of Kryptonate preparations were employed:

a) An ion bombardment preparation utilizing 7075-T6 specimens. Some specimens were prepared with an accelerating voltage of 1.5 kV for one side and 4.5 kV for the other side. However, for most specimens used in this, and later studies, a 1.5 kV accelerating voltage was used. All preparations were made using 27.6% Kr^{85} in Kr supply gas.

b) Diffusion preparations utilizing 7075-T6 specimens. Preparation temperatures of $100^{\circ}C$ were used and preparation times were 10 days to 2 weeks. The pressurizing gas supply was about 75 curies of 5% Kr^{85} in Kr.

c) Diffusion preparations utilizing 7075-O specimens. In this case, the specimens were inserted in a pressure vessel filled with 5% Kr^{85} in Kr. An attempt was then made to duplicate the tempering treatment utilized to convert 7075-O to 7075-T6. This treatment includes a rapid quench which was simulated by immersing the hot, massive pressure vessel containing the specimens in liquid N_2.

3.2 Fatigue Testing

Testing was performed by Teledyne Materials Research Corporation, Waltham, Massachusetts, on the above mentioned Schenk machine which provides for axial loading. All tests reported herein, unless otherwise noted, were performed under the conditions:

Maximum stress	- 47,000 psi
Minimum stress	- 11,750 psi
Mean stress	- 29,375 psi

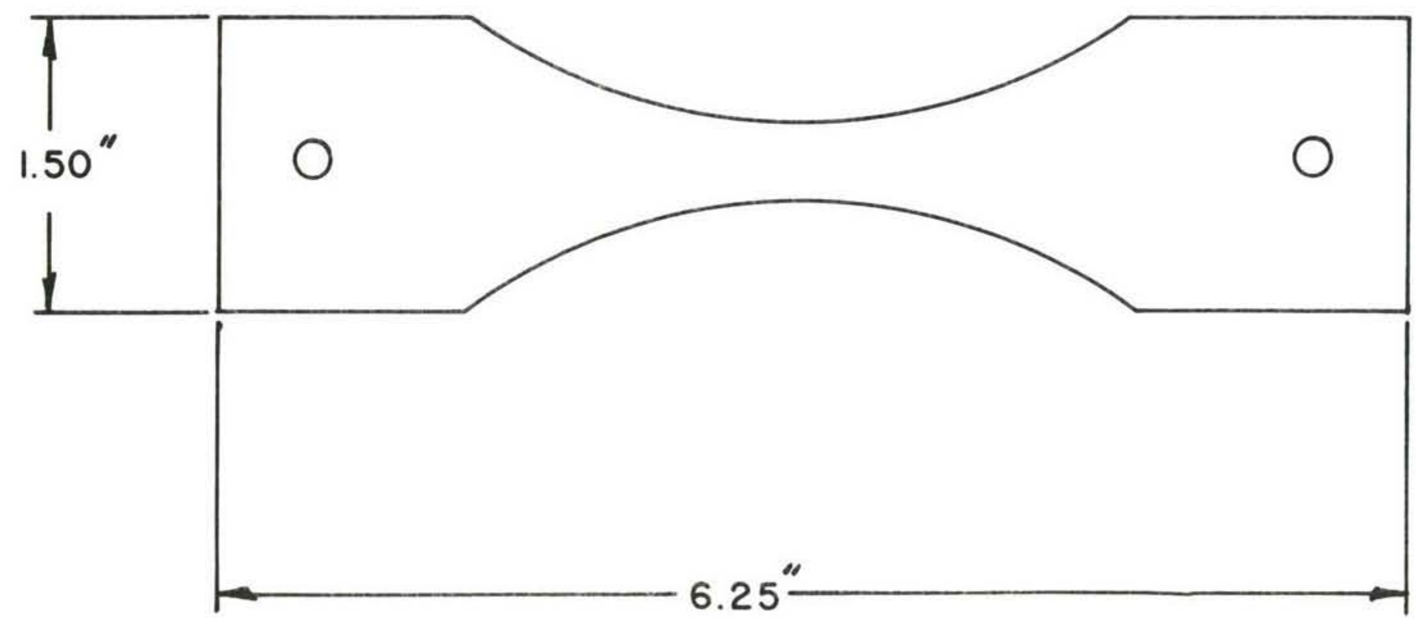


FIG. I. FLAT FATIGUE TESTING SPECIMEN

From Smith's (8) curves for 7075-T6, the expected fatigue life for these conditions is 200,000 cycles.

The Kr^{85} activity level on one side of the specimen was continuously monitored with a Nuclear Chicago GM tube #108 and ratemeter #1620SC. In addition, the testing was interrupted after every 20,000 cycles to obtain a more accurate Kr^{85} level count for both sides of the specimen.

3.3 Results

Fatigue test data was obtained only for those specimens which were prepared from 7075-T6. Those specimens which were fabricated from 7075-O, and for which a tempering treatment to produce 7075-T6 was provided, failed before full tensile load could be applied during the test. Apparently the quench provided was not sufficient to develop T6 mechanical properties.

Kr^{85} loss data obtained for both sides of two specimens, one prepared by ion bombardment and the other by diffusion, are shown in Fig. 2. Radioactive statistical uncertainties corresponding to 3σ (σ = standard deviation) are indicated for each side. Because loss rates were small in all cases, it was thought that greater sensitivity might be obtained by restricting the area viewed. (The GM tube, for the data shown in Fig. 2, viewed a 1" diameter circle centered at the midpoint of the specimen.) Accordingly, samples were notched to ensure fracture at the center line and the GM tube was shielded so that it viewed the entire width of the specimen at the center line but for a distance of only 0.094" on either side of the specimen midpoint. This notched specimen was tested with a maximum stress of 30,000 psi so that its fatigue life was 191,400 cycles.

The data obtained have been treated by assuming the Kr^{85} loss rate to be linear with fatigue cycling according to the equation

$$\%\ \text{activity retained} = 100 - mc$$

where c is the number of cycles and m the rate of Kr^{85} loss. Least square values for m were derived and selected results are shown in Table 1. Also shown therein are values for s_m, the standard deviation of m, and the ratio m/s_m which is a measure of the statistical probability that $m \neq 0$, i.e., if $m/s_m = 2$, then $m > 0$ at the 95% confidence level.

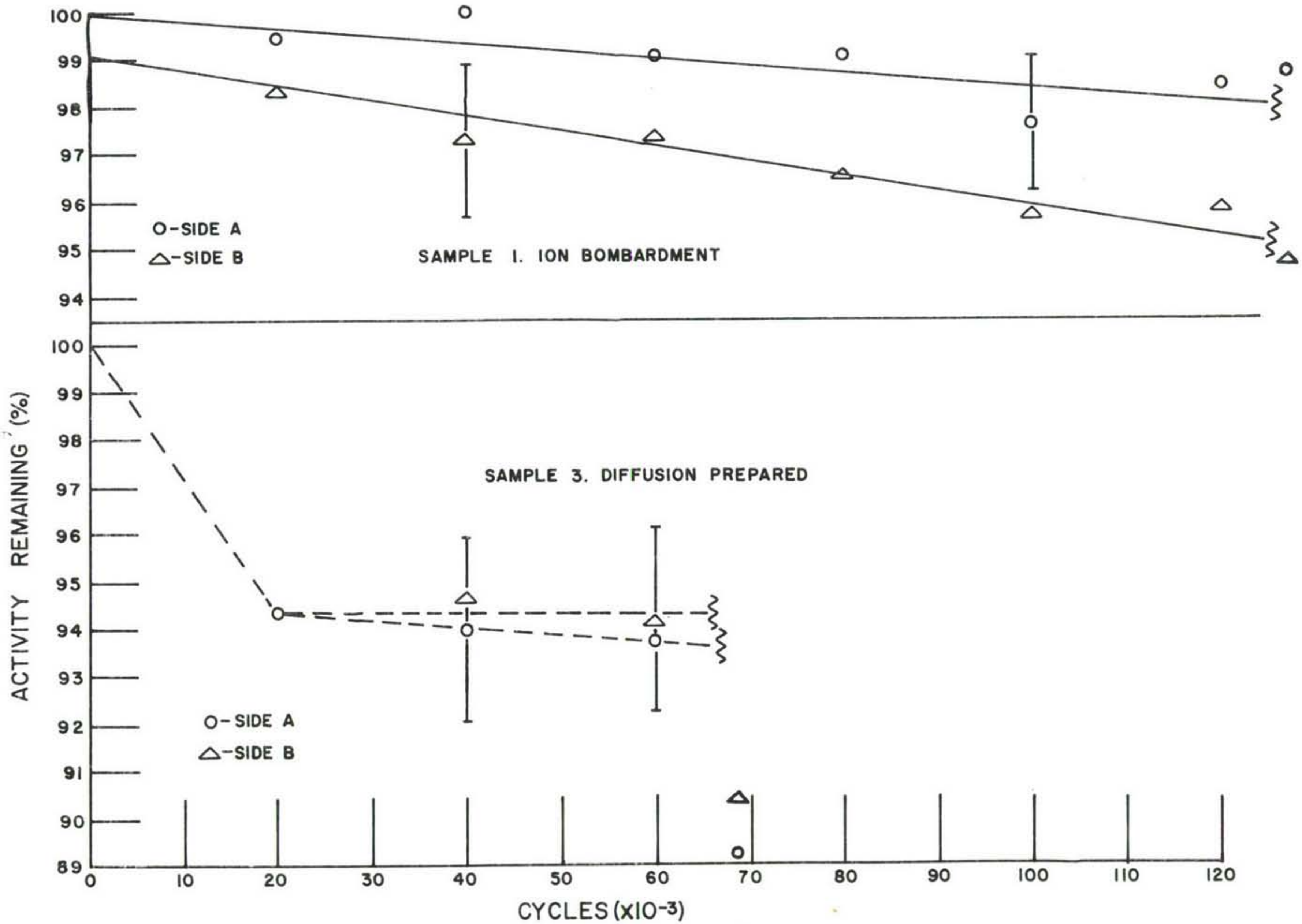


FIG. 2 KR-85 RELEASE DUE TO FATIGUE TESTING

TABLE 1

Least Square Constants for Kr^{85} Loss
During Fatigue Testing

Specimen No.	1		2		3		4	
Specimen Side	A	B	A	B	A	B	A	B
$m \times 10^5$, %/cycle	1.70	3.36	4.52	5.00	9.61	8.74	0.23	0.46
$s_m \times 10^5$	0.46	0.46	0.08	2.45	4.63	3.81	3.8	4.2
m/s_m	3.7	7.4	57.9	2.0	2.1	2.3	0.06	0.11

Note:

Specimens 1 and 2 prepared by ion bombardment
Specimen 3 prepared by diffusion
Specimen 4 prepared by ion bombardment, notched.

******

Autoradiographs were taken of both sides of the specimen at various stages of a test. However, these radiographs were not informative.

Discussion and Conclusions

The data shown in Fig. 2 demonstrate that the loss of Kr^{85} from specimen prepared by diffusion is nonlinear. In fact, other experiments showed that the Kr^{85} loss occurred during the first 5% of the fatigue test. It is therefore not too surprising that the statistical confidence in a linear loss rate for this specimen preparation should be relatively low. However, this Kr^{85} loss behavior showed little promise for utilization as a predictor of fatigue life.

Specimens prepared by ion bombardment (Nos. 1 and 2 in Table 1) showed relatively small loss rates but these rates were linear and, for the most part, statistically significant at a relatively high confidence level. However, the results for notched specimens contradicted the earlier data. If the losses of Kr^{85} for specimens 1 and 2 were associated with fatigue damage that results in ultimate failure of the specimen, then this effect should have been magnified for the notched specimen both because major fatigue damage should occur in the area around the notch and because it was only this area for which Kr^{85} content was monitored. On the contrary, the data indicated an extremely small Kr^{85} loss rate which could not statistically be differentiated from zero.

The conclusion was therefore reached that, whatever the reasons for Kr^{85} loss in specimens 1 and 2, that loss was not associated with fatigue damage. Even if these losses could have been attributed to damage, the rate was much smaller than had been anticipated and the development of a test for fatigue life based upon such loss rates would have been questionable.

Although prior studies involving Kryptonates had shown a high sensitivity to surface disturbances, all such studies involved an actual disruption of the crystalline structure at the surface, either by virtue of a chemical attack at the surface or as a result of permanent physical motion of one crystalline grain with respect to another. During fatigue testing, however, one is careful not to exceed the elastic limit and to eliminate flow. Under such conditions, major disruption of the crystalline structure is not expected. It is believed that the zero, or near zero, Kr^{85} loss rate is associated with the fact that permanent deformation, or flow, does not occur.

Accordingly, it was decided that the validity of a fatigue life test based upon Kr^{85} loss behavior from a fatigue specimen had been established as negative. Subsequent experiments performed during this program were directed towards exploring other phenomena associated with Kryptonates which might be correlatable with remaining fatigue life. These experiments are reported and discussed in the following section.

4. Exploratory Studies

As discussed in the previous section, the simple approach towards the assessment of remaining fatigue life was not fruitful. It was decided to determine whether any other properties of Kryptonates might be relatable to the presence of fatigue damage. One property which suggested itself as being potentially useful is the extremely high sensitivity to surface chemical attack. This sensitivity arises from the fact that the Kr^{85} retained by a solid is situated in the surface layers only at very shallow depths. Thus, release behaviors are representative of phenomena occurring only in the surface layer. It was therefore postulated that areas wherein fatigue damage occurs might have somewhat different energies than adjacent areas. Hence their reactivity to chemical attack would be different and would be revealed by different release rates for Kr^{85}.

Another property of Kryptonates which was thought to be of possible value in the study of fatigue is concerned with the nature of the trapping sites in the metal. As was discussed earlier (see Section 2), Kr^{85} is held both inter- and intragranularly. It was postulated that a redistribution of Kr^{85} between the grains and the grain boundaries might take place during cyclic stressing (a selective release is ruled out by the lack of detectable releases in the tests discussed in Section 3). The redistribution would be revealed by different degassing curves since binding energies and release temperatures are higher for intragranularly held Kr^{85}.

Experiments designed to explore these properties in relation to fatigue damage have been conducted and are discussed in this section.

4.1 Experimental

A set of 10-12 specimens were electropolished at the same time and subsequent treatments were performed at the same time for all members of the set. All Kryptonate preparations were conducted by the ion bombardment procedure (see Section 3 and Reference 2). Other treatments and the sequence thereof provided for each set are detailed in the discussion for that particular set.

Cyclic stressing was performed on the Schenk machine at the stress levels listed in Section 3.2 for all sets except Set 5. For this latter set a Krause Model VSP 150 Variable Fatigue Plate Testing machine was used and the stress conditions were $\pm$ 28,000 psi.

In general, two members of the set were cycled to failure and the average number of cycles was taken as the fatigue life for that set. Pairs of specimens were then cycled to 60% and 90% of their assumed fatigue life (in the case of Set 4, a 75% level was also added). Another pair of specimens, which was not cycled, was retained for reference. The average fatigue life for each set is listed in Table 2.

TABLE 2

Fatigue Life

Set No.	Average Cycles to Failure
1	81,260
2	40,200
3	91,000
4	82,650
5	112,800
6	61,000

******

Autoradiographs of each specimen surface were taken with Kodak DF-57 dental film after Kryptonate preparation and after each subsequent treatment of the specimen. Radiographs were obtained by placing the emulsion in direct contact with the specimen surface. Exposure and development times were adjusted to obtain optimum definition of structural features. Therefore total intensity on the radiographs is not indicative of higher or lower Kr^{85} content when comparison is made from one radiograph to another. However, relative intensity variation within a single radiograph is representative of corresponding variations in Kr^{85} content. Selected radiographs, which are shown in the following figures, are displayed with one end of the specimen up. Radiographs of both sides of the same specimen are displayed as viewed "head-on."

4.2 Chemical Reactivity

A large number of samples were tested. After some preliminary exploration, it was decided to restrict reacting solutions to approximately 0.001 N NaOH and 0.1 N HCl. Both of these solutions are extremely mild reactants and exposure times were kept short - 1-3 minutes. The actual conditions used and their sequencing are listed in Table 3. The sequencing was chosen to explore various possibilities for the application of Kryptonates to fatigue problems. For instance, it was thought that the uptake of Kr^{85} during preparation might be a function of the presence of fatigue damaged areas. Thus, Set 1 was stressed prior to Kryptonate preparation. When the resulting radiographs showed no features that could be associated with fatigue damage, some specimens were then used to develop etching procedures and others were used for the experiments discussed in Section 4.3.

A complete set of radiographs for Set 2 is shown in Fig. 3. It is seen that the distribution of Kr^{85} over the specimen surface as originally prepared is quite uniform even though a few light spots (corresponding to high Kr^{85} concentrations) are evident. Also seen on the 37-B surface are some scratches which were not removed by electropolishing. Radiographs taken after cyclic stressing are substantially unchanged from the unstressed ones, which is consistent with the results discussed in Section 3, i.e., loss of Kr^{85} as a result of cyclic stressing is not detectable.

However, a number of features are displayed by the radiographs taken after a one-minute etch in 0.1 N HCl. Before discussing these, it is well to point out that these features develop as a result of different rates of reactivity with HCl for various portions of the specimen surface. This reactivity is evidenced by an increased rate of Kr^{85} loss from the more reactive areas and is displayed as a dark area in the radiographs depicted in Fig. 3. The most distinctive feature of this set of radiographs

TABLE 3

Sequence of Specimen Treatments

Set #1

A. Cyclic stressing
B. Preparation of Kryptonates
C. Portions etched in 0. 1 N HCl and 0. 001 N NaOH

Set #2

A. Preparation of Kryptonates
B. Cyclic stressing
C. Portions etched in 0. 1 N HCl and 0.001 N NaOH
D. Heated in O_2 to $100^{\circ}C$ for 60 minutes

Set #3

A. Etch in 0.001 N NaOH
B. Preparation of Kryptonates
C. Cyclic stressing
D. Portions restressed to failure

Set #4

A. Etch in 0. 001N NaOH
B. Preparation of Kryptonates
C. Cyclic stressing
D. Etch in 0. 1 N HCl

Set #5

A. Preparation of Kryptonates
B. Cyclic stressing in fully reversed bending
C. Etch in 0. 1 N HCl

Set #6

A. Oxidized in oven at $100^{\circ}C$ for 16 hours
B. Preparation of Kryptonates
C. Cyclic stressing
D. Etch in 0. 1 N HCl

Before Cycling | After Cycling | After Etch

11-T

11-B

12-T

12-B

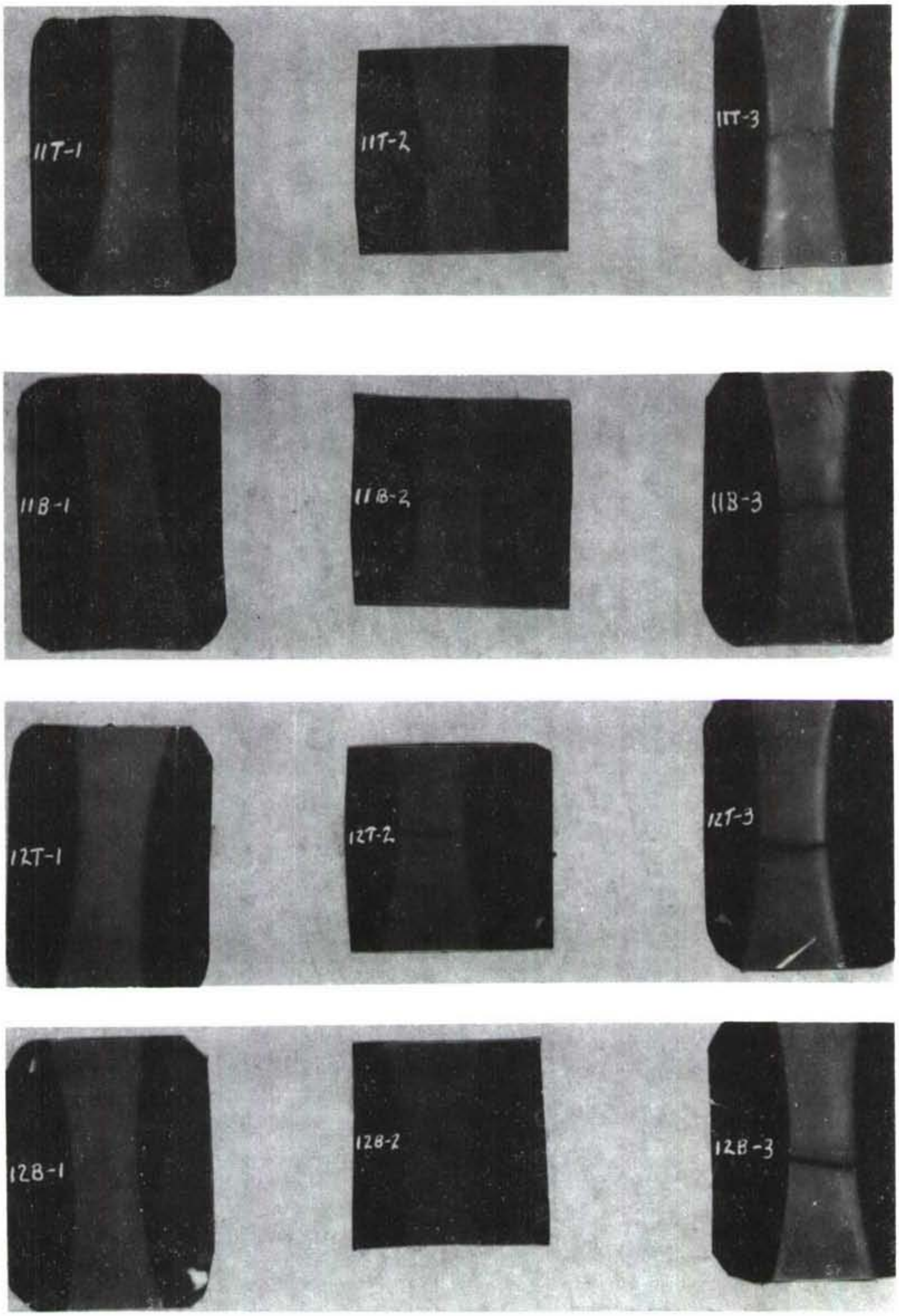


Figure 3A. Autoradiographs of Set 2 Cycled to Failure

Before Cycling | After Cycling | After Etch

38-T

38-B

39-T

39-B

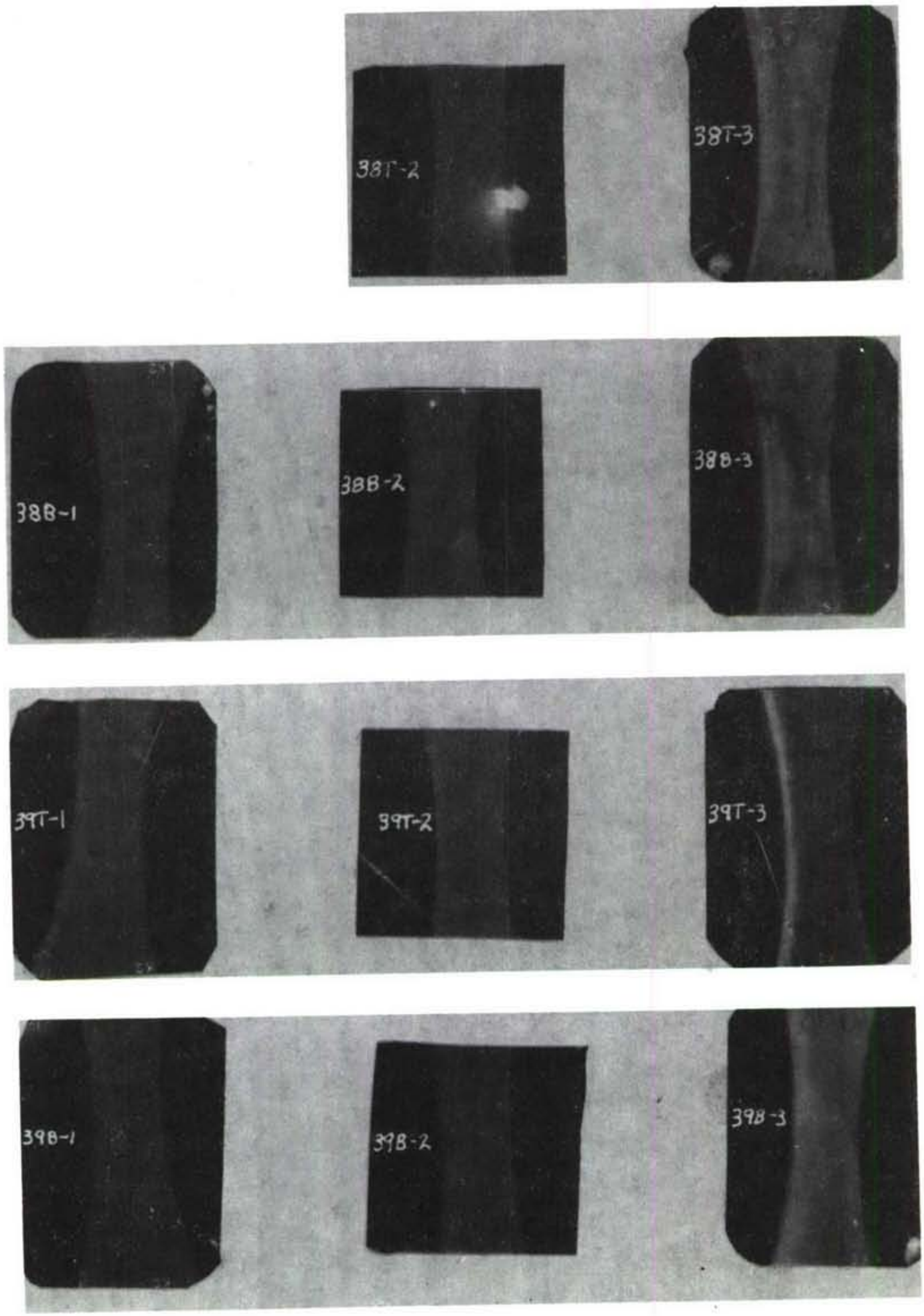


Figure 3B. Autoradiographs of Set 2 Cycled to 90% of Expected Failure

Before Cycling | After Cycling | After Etch

36-T

36-B

37-T

37-B

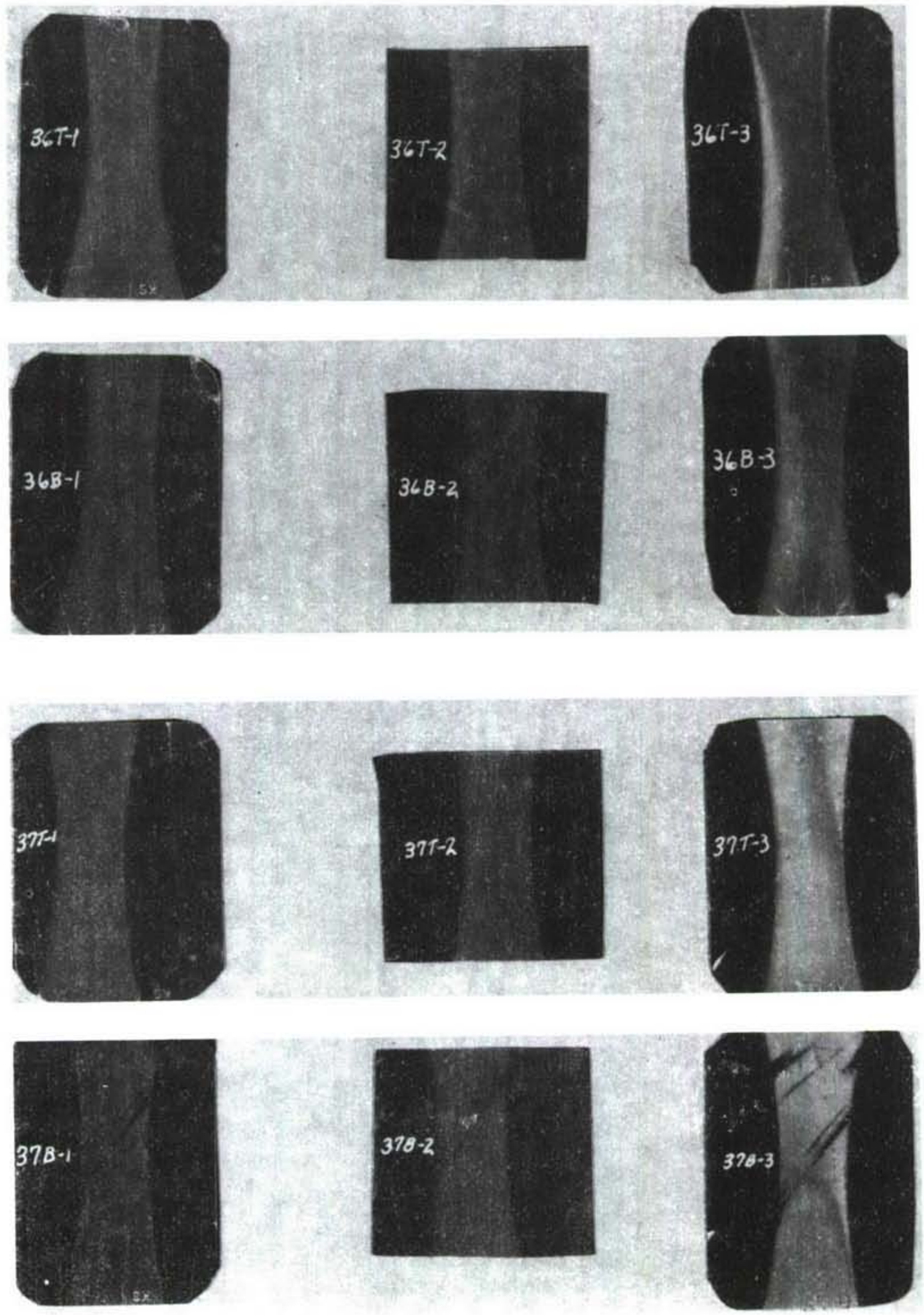


Figure 3C. Autoradiographs of Set 2 Cycled to 60% of Expected Failure

Before Cycling | After Etch

13-T

13-B

40-T

40-B

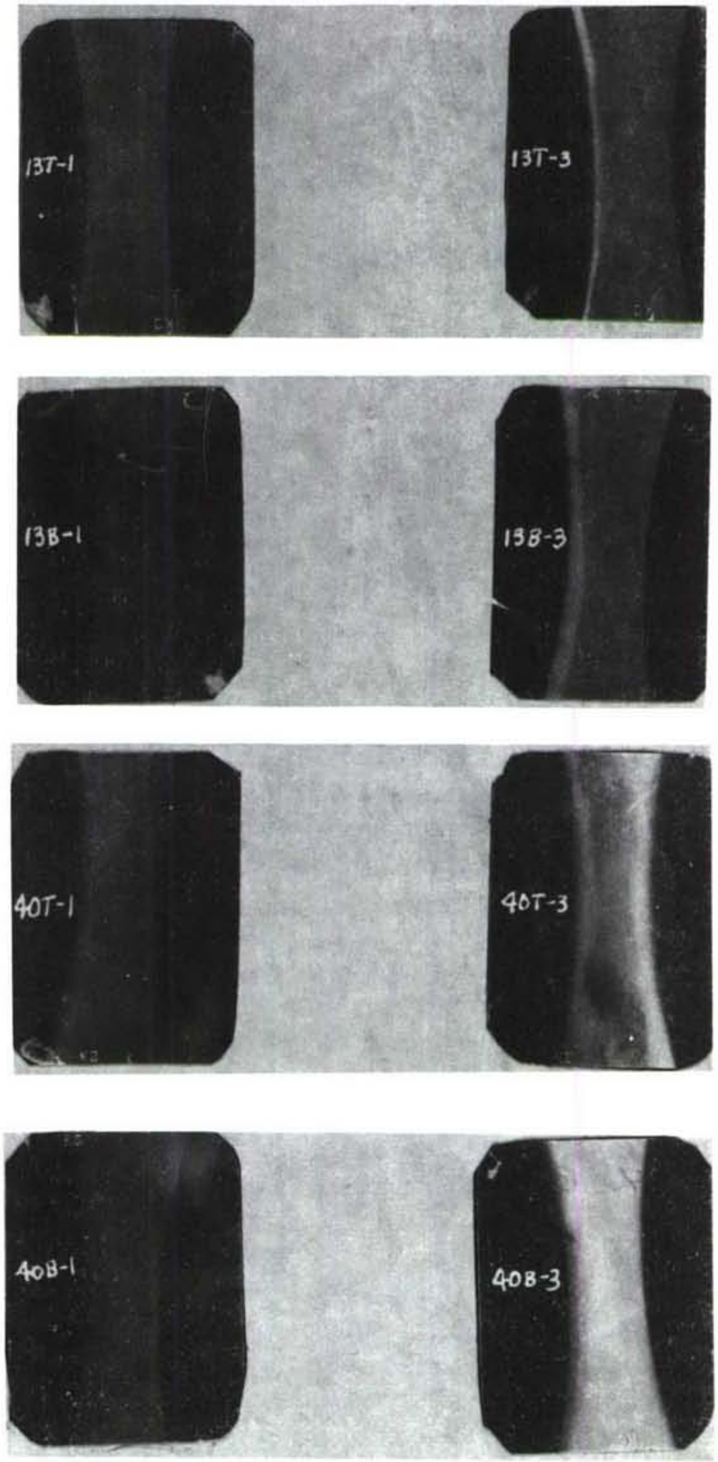


Figure 3D. Autoradiographs of Set 2 Cycled to 0% of Expected Failure (Spares) is shown by surfaces 38-T and B. Both surfaces show considerable differentiation which might be attributed to fatigue damage were it not that a) specimen 39 has undergone the same number of stress cycles but does not exhibit this differentiation, and b) specimen surface 40-T which has not been subjected to cyclic stress shows a similar differentiation. The other noticeable feature of these radiographs is the light ridge running along the edge of many specimens. When attention is drawn to this, it can be seen that such a ridge is present in earlier radiographs but is not as noticeable.

Further experiments to ascertain the origin of the ridges were conducted. It was thought that they might have been caused by the machining operation used to fabricate the specimens. Accordingly, two specimens were deformed by bending about an axis running through the midpoint of the specimen and perpendicular to the specimen length. In one case, (a), the deformation was conducted in the plane of the specimen; in the other case, (b), out of the plane. Both deformations were sufficiently large as to cause flow and permanent deformation. Kryptonates were than prepared and radiographs, shown in Fig. 4, were taken before and after etching in 0. 1 N HCl. Prior to etching no effects attributable to flow can be detected. However, following etching, light areas in the region of flow can be seen on both sides of case (a) even though it is much more prominent on one side than the other. For case (b) a lighter line, corresponding to the line where flow is expected, is seen running across the specimen at its midpoint. Thus, the light ridges found along the specimen edges in the Set 2 radiographs (and in numerous radiographs from subsequent sets) appear attributable to the machining operation.

In Fig. 5, selected radiographs which show features of possible interest are displayed. The striations present on the 15-B surface become more distinct after cyclic stressing to 90% of estimated failure but are similar to those shown on 17-B surface cycled to only 60% of failure. Because it was possible that these individual specimens had the same remaining life (despite the difference in nominal life) all specimens were cycled to failure. The total life for those specimens which were interrupted after 90% of failure was 14-88050 and 15-90900 cycles; for those interrupted after 60% of failure was 16-84200 and 17-68300 cycles. Remarkably good agreement is found between the lives of three of these specimens and the nominal life for this set as listed in Table 2. However, these data indicate that cycling of specimen 17 was interrupted after 80% of its actual life rather than the nominal 60%. Thus, the possibility existed that the more clearly defined striations present on surfaces 15-B and 17-B after 90% and 80% of their respective fatigue lives were related to fatigue damage.

After Deformation

After Etch

26-T

26-B

27-T

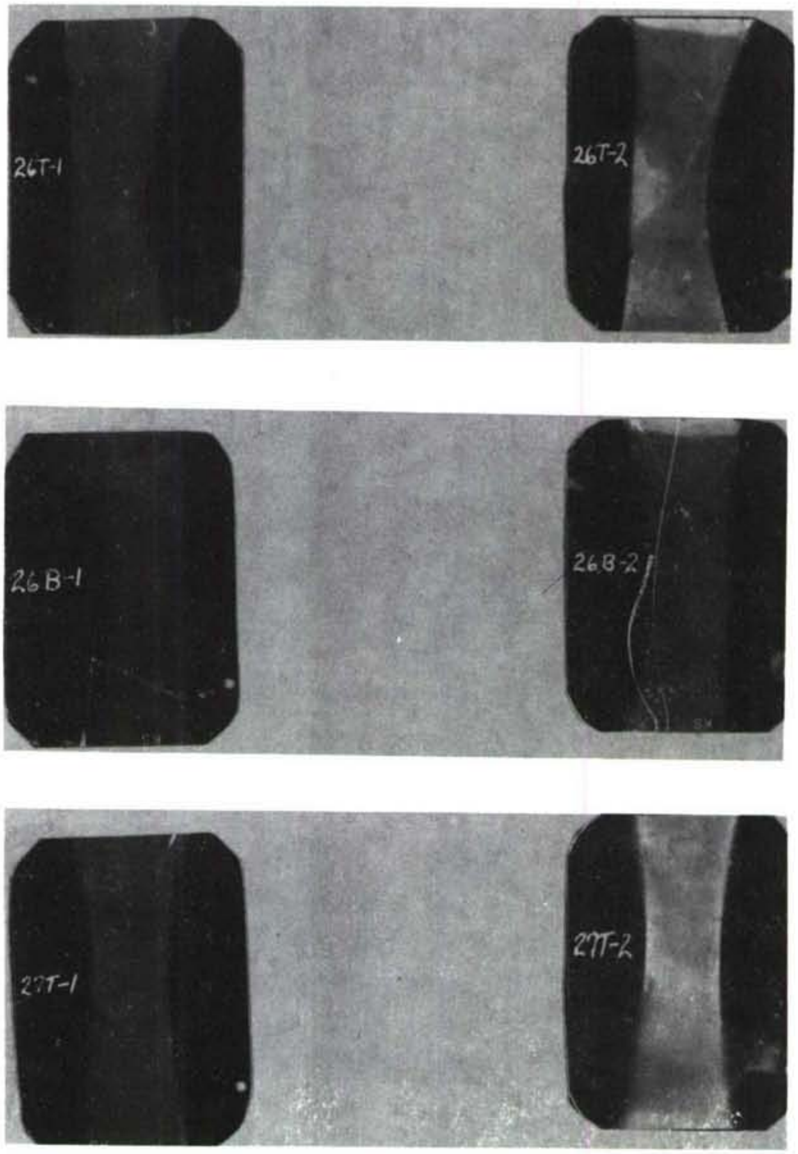


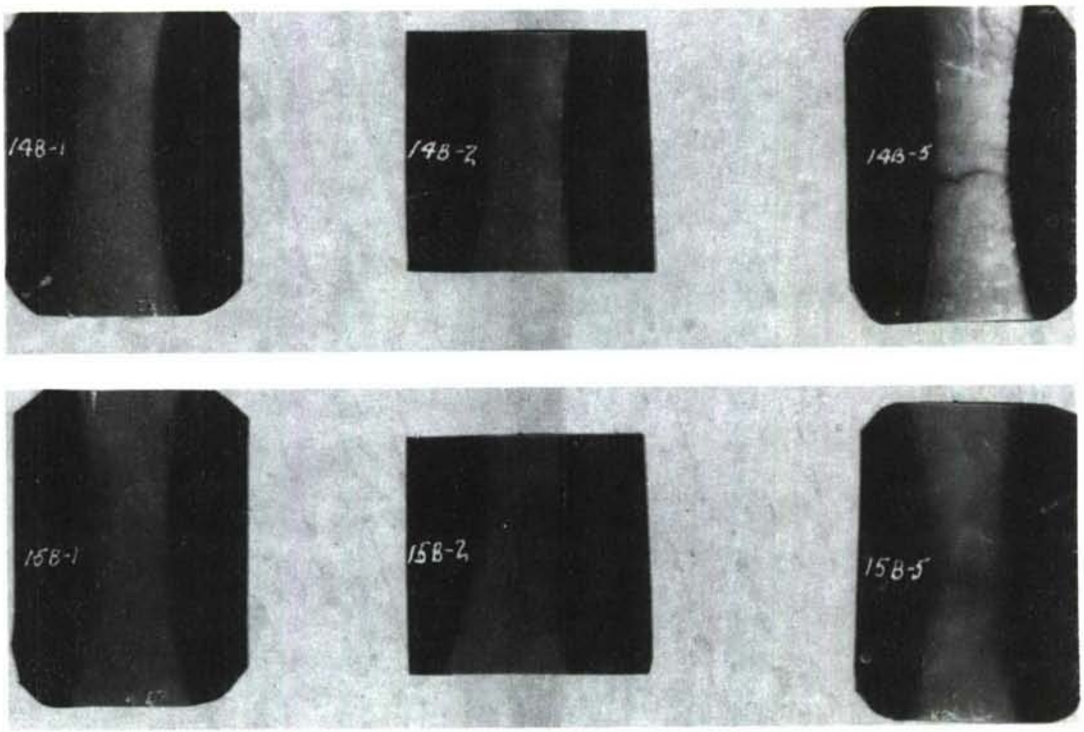
Figure 4. Autoradiographs of Deformed Specimens. Specimen 26: Case (a) deformation; Specimen 27: Case (b) deformation (see text).

Cycled to 90% of Expected Failure

Before Cycling	After Cycling	After Cycling to 100% of Failure

14-B

15-B



Cycled to 60% of Expected Failure

16-B

17-B

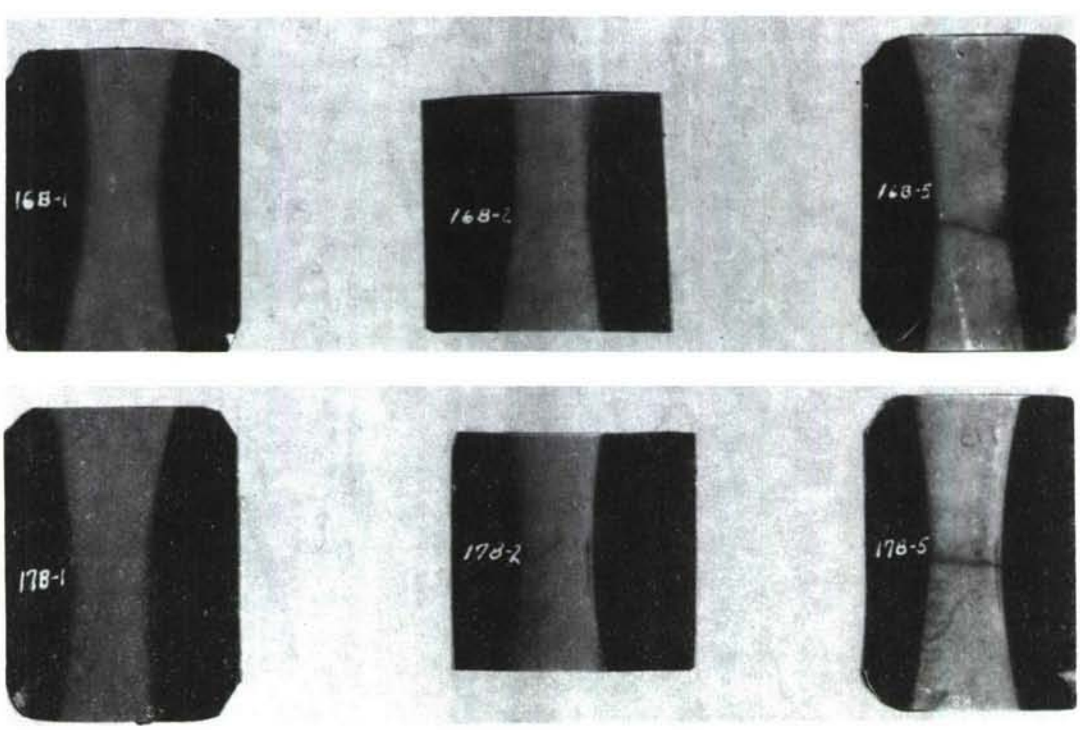


Figure 5. Selected Autoradiographs of Set 3

However, no such striations were found on the opposing surfaces of these specimens nor do any clearly defined striations appear on either the 14-B surface after 93% of its actual life or the 16-B surface after 65% of its actual life. The results, although indicating the possibility of effects associated with the fatigue process, are nevertheless ambiguous.

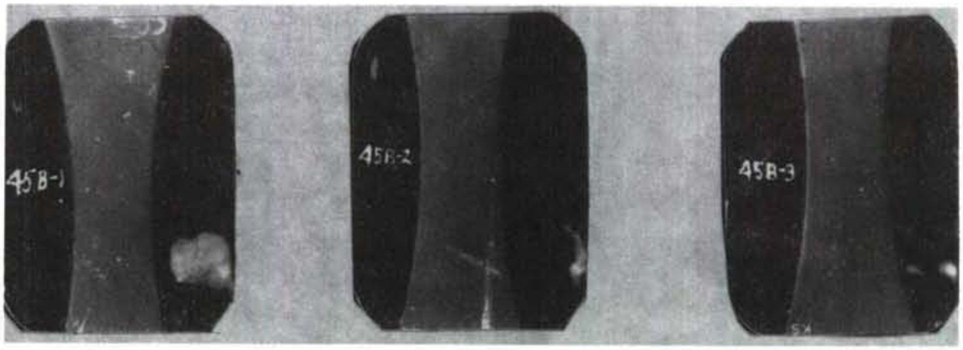
This ambiguity was further investigated by the radiographs obtained from Set 4 which was processed in the same manner as Set 3 prior to testing. For this set no striations were detectable after cycling nor were any other features that could be associated with fatigue damage. Some structure was observed as on surface 46-T (see Fig. 6) but a more frequently encountered radiographic pattern is shown by surface 45-B, also seen in Fig. 6. Both surfaces had been subjected to 75% of their nominal fatigue life.

All sets discussed thus far were tested on the same test machine at the same stress levels. Under these conditions crack propagation followed so closely upon initiation that no partially failed specimens were obtained. To ascertain the effects, if any, of testing conditions, Set 5 was cycled in fully reversed bending at a high stress level. One partially failed specimen was obtained which was cycled to 90% of nominal failure. This is shown by surface 62-T in Fig. 7 wherein a crack, visible to the eye, is indicated by an arrow. The radiograph taken after HCl etch shows a pattern of dark spots. But even in this situation where it is known that failure is imminent, the radiographic pattern cannot be differentiated from that of surface 60-B which has been cycled to only 60% of nominal failure, i. e., the crack is not detectable by the techniques used. If, however, the Kryptonate preparation procedure were to be applied to a partially failed specimen, the crack would become evident in the same manner as the scratch shown in Fig. 3.

Still another possibility that was considered was that the adhesion of an oxide layer on the alloy surface might be different in areas of fatigue damage than in other areas. Accordingly, an oxide layer, as heavy as possible without significantly affecting mechanical properties, was developed and Kryptonates were prepared from these specimens. Except for the presence of the failure crack, the surface of 100-T, shown in Fig. 8, appears the same before and after cycling. The surface of 94-T, which was cycled to 90% of nominal failure, is also shown in Fig. 8 to illustrate the appearance of a Kryptonate prepared from an oxidized surface. The oxide modules retain significantly more Kr^{85} than the metal and yield a "pockmarked" radiograph. It may be parenthetically noted that all Kryptonates prepared during this program by ion bombardment should have significant fractions of the Kr trapped in the alloy itself rather than in the oxide only. Doherty and Davis (9) report that an

Before Cycling | After Cycling | After Etch

45-B



46-T

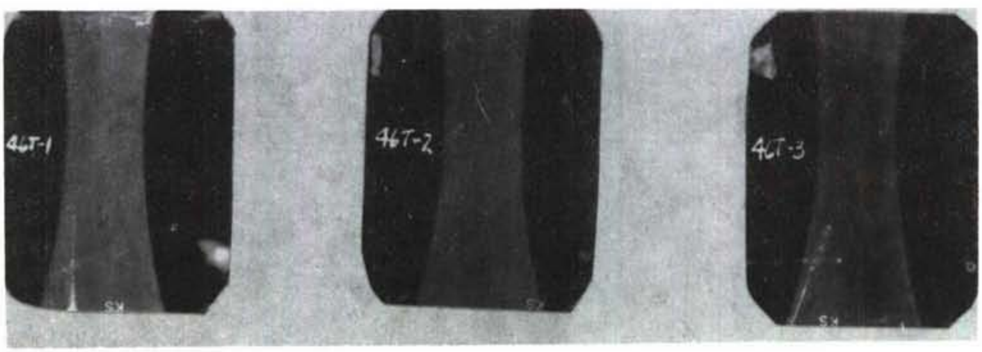
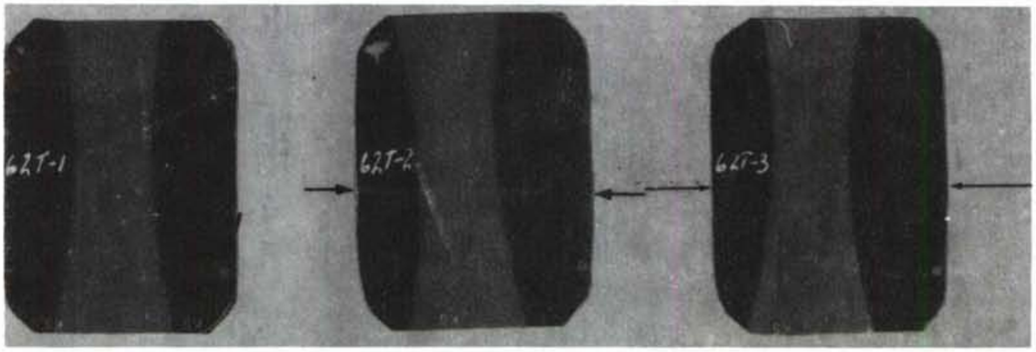


Figure 6. Selected Autoradiographs of Set 4 Cycled to 75% of Expected Failure

Cycled to 90% of Expected Failure

Before Cycling | After Cycling | After Etch

62-T



Cycled to 60% of Expected Failure

60-B

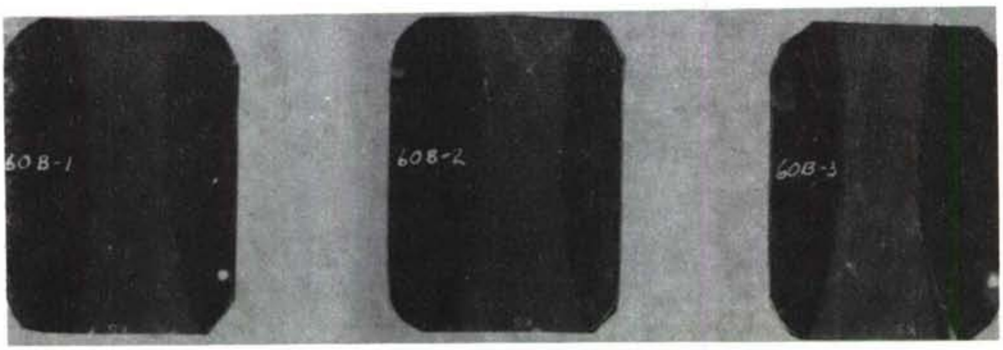


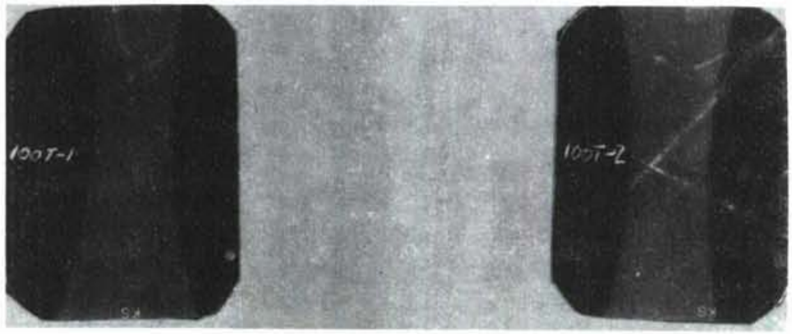
Figure 7. Selected Autoradiographs of Set 5

Cycled to Failure

Before Cycling

After Cycling

100-T



Cycled to 90% of Expected Failure

94-T

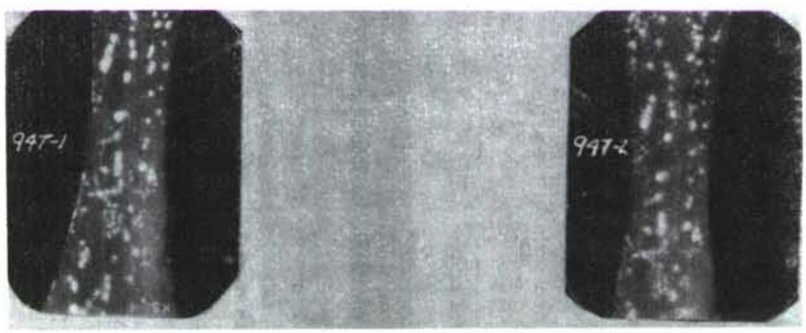


Figure 8. Selected Autoradiographs of Set 6

amorphous oxide film is formed on aluminum below 475°C with a limiting thickness of about 50 angstroms. However, the ion penetration depth ranges upwards from 100 angstroms depending upon the substrate and upon the incident ion energy (10). Thus, Kr^{85} ions should penetrate the extremely thin oxide layer formed by exposure of the specimen to air following electropolishing and the ions then become trapped in the alloy.

To recapitulate the experiments performed which involve chemical reactivity, it may be stated that a number of features have been observed, including the detection of effects attributable to machining operations. Radiographic features have been found which may be associated with fatigue damage but a coherent pattern cannot be deduced. It may be that the statistical fluctuations known to be associated with fatigue specimens and phenomena are masking the effects sought or it may be that the effects of fatigue are subtler than can be revealed by the methods employed. Conversely, the structural patterns found may be some of the precursors of fatigue damage. Much further study would be required to substantiate the radiographic features or to determine that they are artifacts.

4.3 Thermal Release of Kr^{85}

As discussed above (see Section 2), at any constant elevated temperature, a portion of the Kr^{85} trapped by a solid is released. The release soon ceases and the solid becomes stabilized at that temperature. Further release occurs at higher temperature. A degassing curve is determined by plotting the stabilized Kr^{85} content of the solid against the stabilization temperature. Degassing curves were determined for portions of Set 1 specimens (see Table 3). These curves were determined by monitoring the Kr^{85} content of the specimen as it was heated in steps in a vacuum furnace at 10^{-3} to 10^{-4} torr. The resulting degassing curves are shown in Fig. 9. Reproducibility for two specimens cycled to 90% of nominal failure is excellent. It is also seen that the degassing curve shows that the loss of Kr^{85} commences at lower temperatures for specimens that have undergone larger numbers of stress cycles.

These data suggest that a redistribution of Kr^{85} from intragranular trapping sites to grain boundaries occurs as a result of stress cycling. It is known (7) that Kr^{85} held at grain boundaries is released at lower temperatures. This redistribution is further supported by the smaller rate of decrease of the degassing curve with increasing temperature for the specimens cycled to 90% of nominal failure than for those specimens subjected to fewer stress cycles.

Despite the promise of these data, this approach was not pursued further because such testing is destructive. The degassing curve cannot

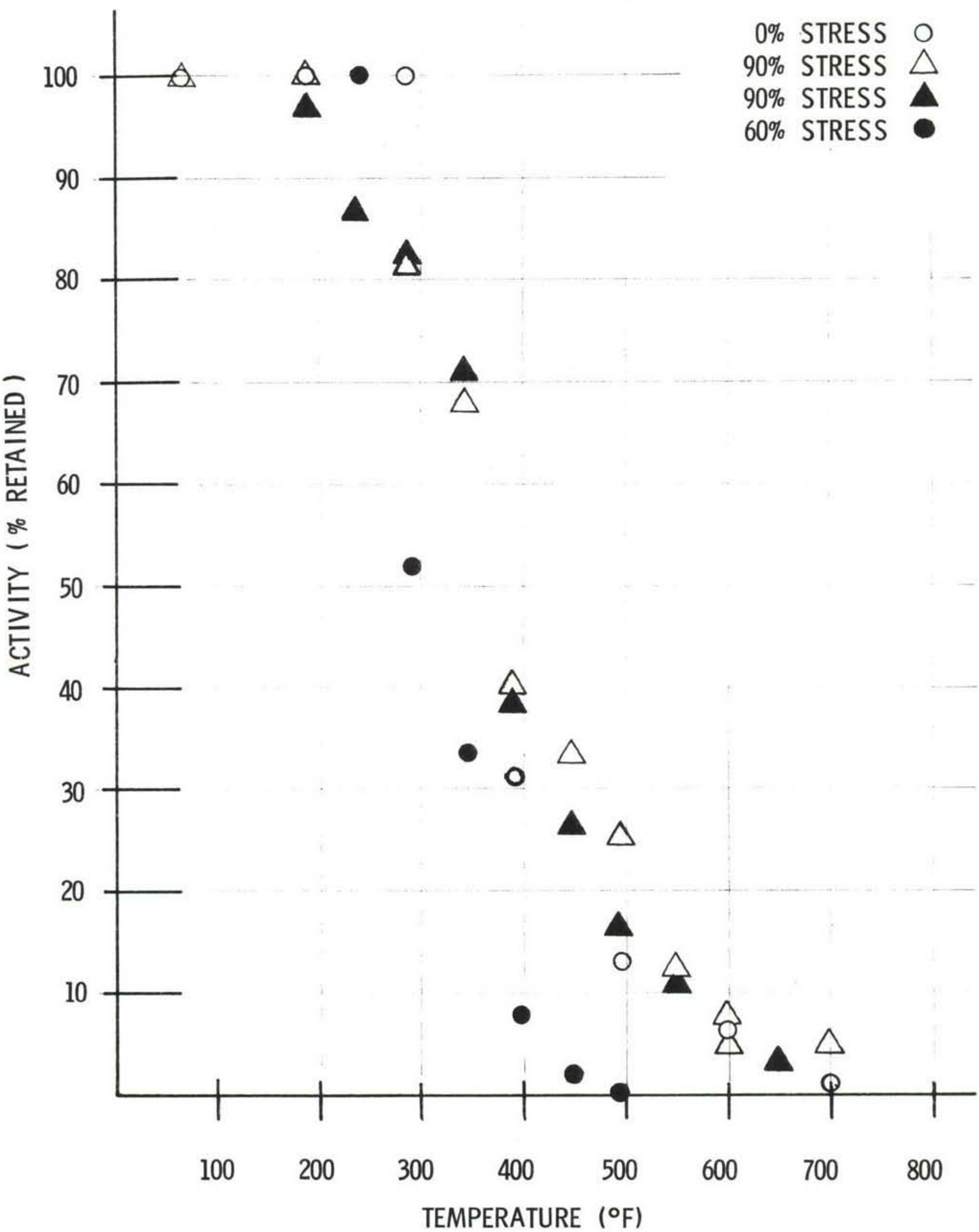


FIG. 9 DEGASSING CURVES

be determined without severely altering the mechanical properties of the specimen. Thus, although further tests might establish the validity of the correlation shown in Fig. 9, the test would not be predictive of future life - the objective of the present program.

5. Conclusions

The utility of the Kryptonate technique for the prediction of future fatigue life of aluminum alloy 7075-T6 has been investigated. The initial approach specified for this program involved measurement of the rate of loss of Kr^{85} from a specimen during fatigue cycling. The rate of loss was found to be very small or zero and the approach to be nonproductive. Alternate methods of utilizing Kryptonates for the study of fatigue damage were investigated. A correlation was found between the onset temperature of Kr^{85} thermal release and the extent of previous stress cycling. However, this measurement results in the alteration of the mechanical properties of the alloy.

The high sensitivity of the Kryptonate technique to surface attack was also utilized but with ambiguous results. Very mild chemical reaction at the surface of a fatigue specimen showed that regions of differing reactivity could be detected. Effects attributable to machining operations during specimen fabrication were demonstrated. Numerous structural patterns were revealed by this Kryptonate etching procedure but no coherent pattern that could be directly related to fatigue damage has been deduced. These patterns may indeed be related to fatigue damage and failure but, if so, the relation is more subtle and indirect than can be revealed by the experiments performed.

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ADDENDUM

Following preparation of this report, a report of a similar study was received. N. A. Tiner and S. K. Asunmaa (Microautoradiography of Kryptonated Aluminum Alloys, presented at ASTM Electron Microscope Committee E-4 Meeting, San Francisco, California, June 1968; Materials Research and Standards, to be published) used 7075-T6 aluminum alloy fatigue specimens into which Kr^{85} was introduced by diffusion. Surface structure and Kr^{85} distribution were examined by microautoradiography which involved electron microscopy of superimposed autoradiographs and surface replicas. Their studies showed a negligible loss of Kr^{85} from the surface of a specimen during the first 1% of fatigue life. However, migration of Kr^{85} along slip markings was detected. A 37.5% loss of Kr^{85} from a fatigue specimen was found following failure and was accompanied by both a segregation of Kr^{85} in the fissure interfaces and a depletion of Kr^{85} in zones near the fissure.

Tiner and Asunmaa's technique of microautoradiography yields information on a microscopic scale but the results obtained are generally in accord with the results reported here. As opposed to the negligible loss reported by Tiner and Asunmaa after less than 1% of fatigue life, a small but measurable loss of about 6% of the Kr^{85} content was found after 20% of fatigue life for diffusion prepared specimens. No further loss was observed during cycling until failure occurred whereupon a sudden loss to about 10% was found which is to be compared to T & A's 37.5% loss. The general behaviors are quite similar and the differences in actual percentage losses may be attributable to actual specimen variability as well as differences in Kryptonate preparation procedures.

Moreover, the redistribution of Kr^{85} among various surface trapping sites which has been postulated here on the evidence of degassing curves is supported by T & A's observation of Kr^{85} migration along slip markings and of Kr^{85} segregation in fissure interfaces. It should be noted that our results were obtained from specimen Kryptonates prepared by ion bombardment, whereas T & A's specimens were prepared by diffusion.

Only one area of partial disagreement exists which concerns T & A's conclusion that microautoradiography is a useful tool for evaluating different stages of fatigue damage. If Kr^{85} migration occurs only in the initial stages of fatigue cycling and at failure, as is indicated by both T & A's data and this data for a diffusion prepared specimen, then it would appear difficult to detect and characterize specimens with 10-90% of total life remaining. On the other hand, the degassing curves for ion bombarded specimens indicate a redistribution occurring continuously throughout fatigue life.

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13. ABSTRACT

The behavior during stress cycling of a Kryptonate(R), prepared from aluminum alloy 7075-T6 fatigue specimens, was studied. Kryptonates are solids into the surface of which the radioisotope, Kr^{85}, has been stably incorporated. They release Kr^{85} at a rate dependent upon the rate at which the surface is disturbed either physically or chemically. Their preparation and properties are discussed.

Efforts to determine whether the release rate of Kr^{85} from a fatigue specimen would be a predictor of remaining fatigue life are reported. Other properties of krypton impregnated specimens, such as high sensitivity to chemical attack and Kr^{85} thermal release behavior were investigated in relation to the fatigue process.

A correlation was found between the onset temperature of Kr^{85} thermal release and the extent of previous stress cycling. The validity of a fatigue life test based upon Kr^{85} loss behavior from aluminum alloy 7075-T6 fatigue specimens was established negatively.

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